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IS 11255-7 (2005): Methods for measurement of emission from stationary sources, Part 7: Oxides of nitrogen [CHD 32: Environmental Protection and Waste Management]



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स्थिर स्रोतों से उत्सर्जन को मापने की पद्धति  
भाग 7 नाइट्रोजन के ऑक्साइड

*Indian Standard*  
METHODS FOR MEASUREMENT OF EMISSION  
FROM STATIONARY SOURCES  
PART 7 OXIDES OF NITROGEN

ICS 13.040.40

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**BUREAU OF INDIAN STANDARDS**  
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## FOREWORD

This Indian Standard (Part 7) was adopted by the Bureau of Indian Standards after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Nitrogen oxides ( $\text{NO}_x$ ) the term is used to describe the total  $\text{NO}$ ,  $\text{NO}_2$  and other oxides of nitrogen are considered important due to their role in the formation of ozone (a secondary air pollutant) through a complex series of petrochemical reactions involving volatile organic compounds.  $\text{NO}_x$  are emitted as part of most fuel combustion process, with nitric oxide ( $\text{NO}$ ) being a primary constituent. The majority (95 percent) of nationwide  $\text{NO}_x$  that is produced from fuel combustion sources is  $\text{NO}$  with the remaining fraction being predominantly  $\text{NO}_2$ .

Exposure to nitrogen dioxide occurs; through the pulmonary system.  $\text{NO}_2$  irritates the lungs. Asthmatics are more sensitive to the effect of  $\text{NO}_2$  than the general population. Exercise increases the ventilation rate and hence exposure to  $\text{NO}_2$ . Some studies have shown a relationship between indoor  $\text{NO}_2$  exposures and increased respiratory illness in young children, short-term  $\text{NO}_2$  exposures ( $< 3$  h duration) at or near ambient concentration levels for some communities in the United States have resulted in an increased respiratory illness in children between 5 and 12 years old, and airway responsiveness/pulmonary function changes in those individuals with pre-existing respiratory illnesses. Long-term exposure levels have been associated with increased susceptibility to respiratory infections and cause lung alterations.

There is no ISO standard on this subject. This standard is developed based on the indigenous technology available in India.

The composition of the Committee responsible for the formulation of this standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

## *Indian Standard*

# METHODS FOR MEASUREMENT OF EMISSION FROM STATIONARY SOURCES

## PART 7 OXIDES OF NITROGEN

### 1 SCOPE

This standard (Part 7) prescribes the methods for determination of oxides of nitrogen from stationary sources.

### 2 PRINCIPLE AND APPLICABILITY

#### 2.1 Principle

A grab sample is collected in a dilute sulphuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulphonic acid (PDS) procedure.

#### 2.2 Applicability

This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams  $\text{NO}_x$  (as  $\text{NO}_2$ ) per standard cubic metre, without having to dilute the sample.

### 3 APPARATUS

**3.1 Sampling Apparatus** — The assembled sampling apparatus is shown in Fig. 1. It consists of the following parts.

**3.1.1 Probe** — Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose).

#### NOTES

1 Stainless steel or Teflon tubing may also be used for the probe.

2 Heating is not necessary if the probe remains dry during the purging period.

**3.1.2 Collection Flask** — Two-litre borosilicate round bottom flask, with short neck and 24/40 standard taper opening, protected against implosion or breakage.

**3.1.3 Flask Valve** — T-bore stopcock connected to a 24/40 standard taper joint.

**3.1.4 Temperature Gauge** — Dial-type thermometer, or other temperature gauge, capable of measuring  $1^\circ\text{C}$  intervals from  $-5$  to  $50^\circ\text{C}$ .

**3.1.5 Vacuum Line** — Tubing capable of withstanding a vacuum of 75 mm Hg absolute pressure, with 'T' connection and T-bore stopcock.

**3.1.6 Vacuum Gauge** — U-tube manometer, 1 m high, with 1 mm divisions, or other gauge capable of measuring pressure to within  $\pm 2.5$  mm Hg.

**3.1.7 Pump** — Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg absolute.

**3.1.8 Squeeze Bulb** — One way.

**3.1.9 Stopcock**

**3.1.10 Barometer** — Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg.

**3.2 Porcelain Evaporating Dishes** — 175 to 250 ml capacity with lip for pouring, one for each sample and each standard.

**3.3 Steam Bath** — Low temperature ovens or thermostatically controlled hot plates kept below  $70^\circ\text{C}$ .

**3.4 Policeman Porcelain** — One for each sample and each standard.

**3.5 Graduated Cylinder** — 100 ml with 1 ml divisions.

**3.6 Spectrophotometer** — To measure absorbance at 410 nm.

**3.7 pH Paper**

**3.8 Analytical Balance** — Accuracy 0.1 mg.

### 4 REAGENTS

**4.1 Absorbing Solution** — To prepare the absorbing solution, cautiously add 2.8 ml concentrated  $\text{H}_2\text{SO}_4$  to 1 litre of distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution.

NOTE — The absorbing solution should be used within 1 week of its preparation. Do not expose to extreme heat or direct sunlight.

**4.2 Sodium Hydroxide (1 N)** — Dissolve 40 g NaOH in distilled water and dilute to 1 litre.

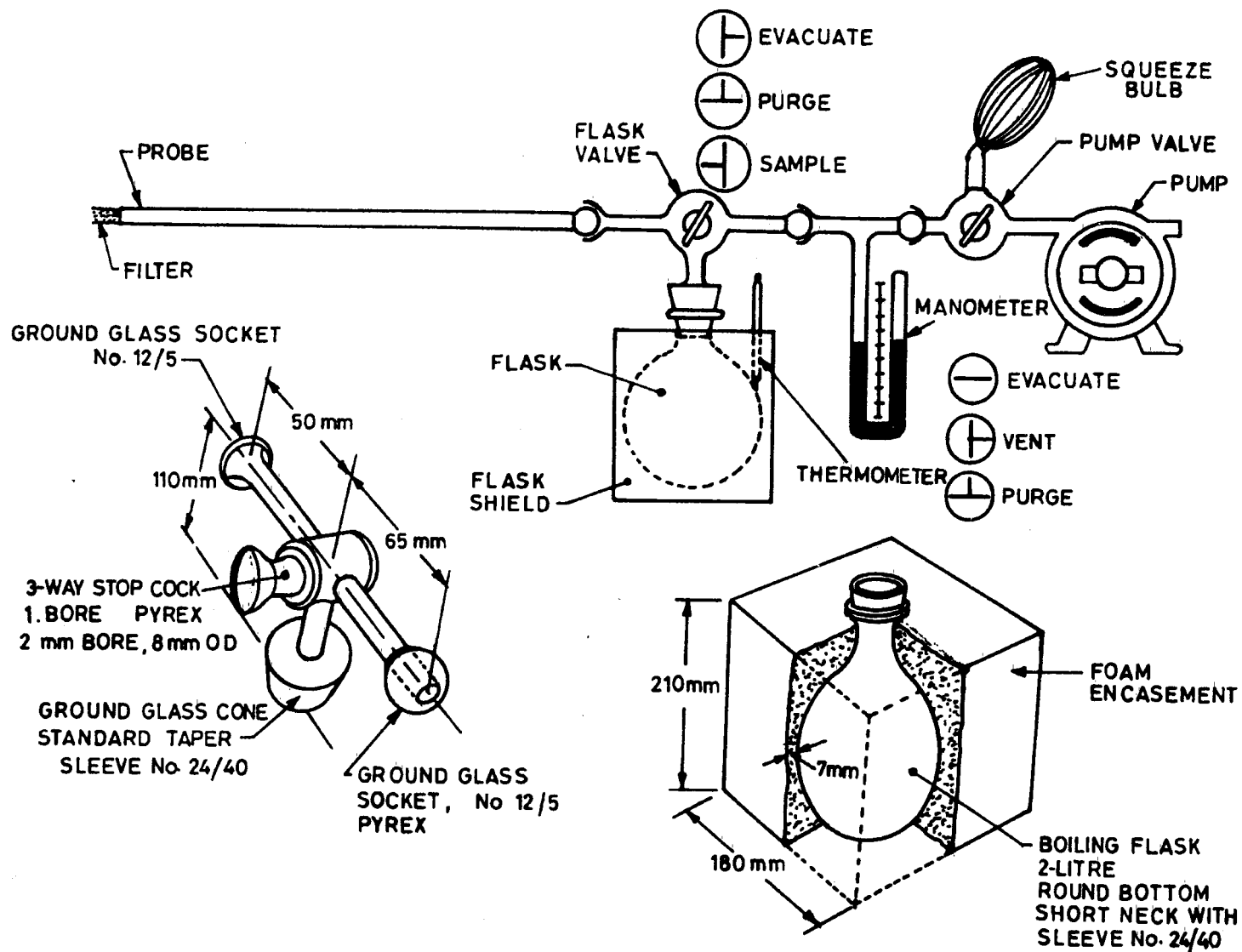


FIG. 1 SAMPLING TRAIN, FLASK VALVE AND FLASK

**4.3 Fuming Sulphuric Acid**, 15 to 18 percent by weight free sulphur trioxide.

NOTE — Highly corrosive, handle with care.

**4.4 Phenol**, white solid.

**4.5 Sulphuric Acid**, concentrated 95 percent minimum assay.

NOTE — Highly corrosive, handle with care.

**4.6 Potassium Nitrate** — Dried at 105° to 110°C for a minimum period of 2 h just prior to preparation of standard solution.

**4.7 Standard KNO<sub>3</sub> Solution** — Dissolve 2.198 g dried potassium nitrate (KNO<sub>3</sub>) in distilled water and dilute to 1 litre with distilled water in a 1 000 ml volumetric flask.

**4.8 Working Standard KNO<sub>3</sub> Solution** — Dilute 10 ml of the standard solution to 100 ml with distilled water. One millilitre of the working standard solution is equivalent to 100 µg nitrogen dioxide (NO<sub>2</sub>).

**4.9 Phenoldisulphonic Acid Solution** — Dissolve 25 g of pure white phenol in 150 ml concentrated sulphuric acid on a steam bath. Cool, add 75 ml fuming sulphuric acid, and heat at 100°C for 2 h. Store in a dark, stoppered bottle.

## 5 PROCEDURE

### 5.1 Sampling

**5.1.1** Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the 'purge' position. Assemble the sampling train as shown in Fig. 1 and place the probe at the sampling point. Make sure that all fittings are tight and leak-free and that all ground glass joints have been properly greased with a high vacuum, high temperature chlorofluorocarbon based stopcock grease. Turn the flask valve and the pump valve to their 'evacuate' position. Evacuate the flask to 75 mm Hg absolute pressure, or less. Evacuation to a pressure approaching the vapour pressure of water at the existing temperature is desirable. Turn the pump valve to its 'vent' position and turn off the pump. Check for leakage by observing the manometer for any pressure fluctuation. Any variation greater than 10 mm Hg over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg absolute at the time sampling is commenced. Record the volume of the flask and valve ( $V_f$ ), the flask temperature ( $T_f$ ) and the barometric pressure. Turn the flask valve counterclockwise to its 'purge' position and do the same with the pump valve.

Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask area, heat the probe and purge until the condensation disappears. Next, turn the pump valve to its 'vent' position. Turn the flask valve clockwise to its 'evacuate' position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask ( $P_f$ ) is equal to the barometric pressure less the manometric reading. Immediately turn the flask valve to the 'sample' position and permit the gas to enter the flask until pressures in the flask and sample line (that is duct, stack) are equal. This will usually require about 15 s; a longer period indicates a 'plug' in the probe, which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its 'purge' position and disconnect the flask from the sampling train. Shake the flask for at least 5 min.

**5.1.2** If the gas being sampled contains insufficient oxygen for the conversion of NO to NO<sub>2</sub> then oxygen shall be introduced into the flask by one of the following three methods:

- Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to 75 mm Hg absolute pressure less;
- Inject oxygen into the flask after sampling; or
- Terminate sampling with minimum 50 mm Hg vacuum remaining in flask, record the final pressure and vent the flask to atmosphere until the flask pressure is equal to the atmospheric pressure.

### 5.1.3 Sample Recovery

Let the flask set for a minimum of 16 h and then shake the contents for 2 min. Connect the flask to mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature ( $T_f$ ), the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask ( $P_f$ ) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5 ml portions of distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1 N), dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into solution and then touching to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid



level so that the container can be checked for leakage after transport. Label the container to clearly identify its contents.

## 5.2 Analysis

Note the level of the liquid in container and confirm whether or not any sample was lost during shipment; note this on analytical data sheet. Immediately prior to analysis, transfer the contents of the shipping container to a 50 ml volumetric flask and rinse the container twice with 5 ml portions of distilled water. Add the rinse water to the flask and dilute to the mark with distilled water; mix thoroughly. Pipette a 25 ml aliquot into the porcelain evaporating dish. Return any unused portion of the sample to the polyethylene storage bottle. Evaporate the 25 ml aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenoldisulphonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all residue. Add 1 ml distilled water and four drops of concentrated sulphuric acid. Heat the solution on a steam bath for 3 min with occasional stirring. Allow the solution to cool, add 20 ml distilled water, mix well by stirring and add concentrated, ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, these shall be removed by filtration as follows:

Filter through Whatman No. 41 or equivalent into a 100 ml volumetric flask, rinse the evaporating dish with three 5 ml portions of distilled water; filter these three rinses.

Wash the filter with at least three 15 ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with distilled water. If the solids are absent, transfer the solution directly to the 100-ml volumetric flask and dilute to the mark with distilled water. Mix the contents of the flask thoroughly and measure the absorbance at the 410 nm wavelength used for the standards using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of distilled water.

## 6 DETERMINATION OF CALIBRATION FACTOR ( $K_c$ )

Add 0 ml, 2 ml, 4 ml, 6 ml and 8 ml of  $\text{KNO}_3$  working standard solution (1 ml = 100 g of  $\text{NO}_2$ ) to a series of fine 50-ml volumetric flasks. To each flask, add 25 ml absorbing solution, 10 ml distilled water and sodium hydroxide (1 N) drop wise until the pH is between 9 and 12 (about 25 to 35 drops each). Dilute to mark with distilled water. Mix thoroughly and pipette 25 ml

aliquot of each solution into a separate porcelain evaporating dish. Beginning from evaporation step follow the analysis procedure as given in 5.2 until the solution has been transferred to the 100-ml volumetric flask and dilute to mark. Measure the absorbance of each solution at 410 nm wavelength on a spectrophotometer. Plot the absorbances of the solution as ordinates against the concentration. A linear relationship is obtained. Calculate the calibration factor ( $K_c$ ) by taking the reciprocal of the slope of the line.

## 7 CALCULATION

### 7.1 Sample Volume Corrected to Standard Condition

Carry out the calculation as given below:

$$V_{sc} = (T_{std}/P_{std}) (V_r - V_a) (P_f/T_f - P_i/T_i) = K_i(V_r - 25) (P_f/T_f - P_i/T_i)$$

where

- $P_f$  = final absolute pressure of flask, mm Hg;
- $P_i$  = initial absolute pressure of flask, mm Hg;
- $P_{std}$  = standard absolute pressure, 760 mm Hg;
- $T_f$  = final absolute temperature of flask, K;
- $T_i$  = initial absolute temperature of flask, K;
- $T_{std}$  = standard absolute temperature of flask, 298.15 K;
- $V_{sc}$  = sample volume at standard conditions, ml;
- $K_i$  = 0.392 3 K/mm Hg;
- $V_r$  = volume of flask and valve, ml; and
- $V_a$  = volume of absorbing solution, 25ml.

### 7.2 Sample Concentration Corrected to Standard Conditions

Carry out the calculations as given below:

$$C = \frac{(A_s - A_b) \times K_c \times 1\,000 \times 2 \times F}{V_{sc}}$$

where

- $A_s$  = absorbance of the sample;
- $A_b$  = absorbance of the blank;
- $C$  = concentration of  $\text{NO}_x$  as  $\text{NO}_2$ , corrected to standard conditions,  $\text{mg}/\text{Nm}^3$ ;
- $F$  = dilution factor (that is, 25/5, 25/10, etc) required only, if sample dilution was needed to reduce the absorbance into the range of calibration;
- $K_c$  = spectrophotometer calibration factor; and
- 2 = 50/25 the aliquot factor.

NOTE— If other than 25 ml aliquot is used for analysis, the factor 2 must be replaced by corresponding factor.

## ANNEX A

## (Foreword)

## COMMITTEE COMPOSITION

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